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Research Paper

Development of Palm Oil-Based Synergist Liquid Membrane Formulation for Silver Recovery from Aqueous Solution

Norela Jusoh [1], Norasikin Othman [1,2,*], Raja Norimie Raja Sulaiman [1,2], Norul Fatiha Mohamed Noah [1], Khairul Sozana Nor Kamarudin [1], Muhammad Abbas Ahmad Zaini [1,2], Dilaeleyana Abu Bakar Sidik [3]

[1] School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
[2] Centre of Lipids Engineering and Applied Research (CLEAR), Ibnu Sina Institute for Scientific and Industrial Research (ISI-SIR), Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
[3] Department of Science and Mathematics, Centre of Diploma Studies, Universiti Tun Hussein Onn Malaysia, 84600 Muar, Johor, Malaysia

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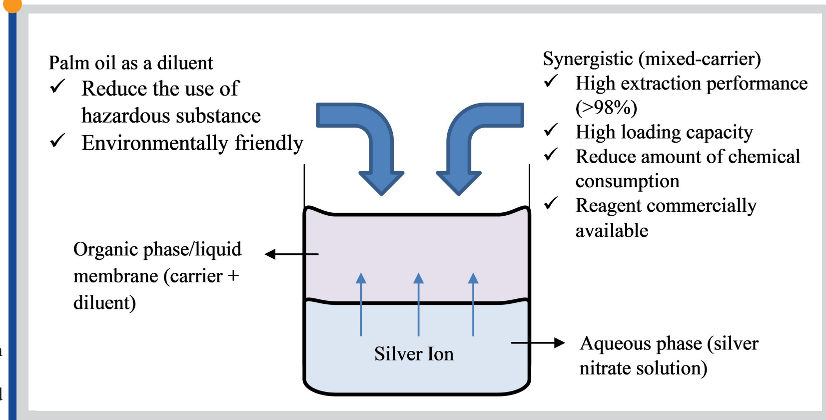
Highlights

- Use of vegetable oil-based diluent to substitute petroleum-based one
- Mixture of carriers to produce a combined effect on extraction performance
- More than 98% of silver ion from a very dilute solution was extracted successfully
- A simple and economic process, as well as contributing to sustainable production

Graphical abstract



Abstract

In this work, the palm oil-based synergist formulation containing a mixture of carriers for liquid membrane application of silver recovery from aqueous solution was studied. Several types of acidic carrier mixtures were investigated via liquid-liquid extraction procedure to increase the extraction performance. The results indicated that palm cooking is a promising vegetable oil-based green diluent in the formulation. A carrier mixture of 0.2 mM Cyanex 302 and 0.3 mM Cyanex 272 demonstrated synergism during the extraction process. It was found that more than 98% of silver ions from a very dilute solution was effectively extracted with a synergistic coefficient of 62.7. According to the individual and carrier mixture results, it can be deduced that Cyanex 272 and Cyanex 302 served as a carrier and synergist, respectively. Therefore, the developed liquid membrane formulation showed great prospect in silver recovery from the liquid waste solution.

1. Introduction

Nowadays, the world faces serious environmental problems, particularly in terms of water pollution due to domestic and industrial discharges. Industrial effluents are usually loaded with toxic heavy metals such as zinc, lead, mercury, silver, and chromium that are harmful to the environment and living organisms. Exposure to these metals over certain concentrations can result in a severe malfunction of the reproduction system, kidney, brain, liver, and central nervous systems of the human body [1]. Hence, numerous works have been conducted to remove toxic heavy metals from wastewater. The electroplating industry is known to be one of the major sources of heavy metals due to the generation of large amounts of wastewater from the rinse water of the electroplated parts [2]. In some electroplating industries, precious metals such as silver are also involved. Therefore, a favorable wastewater treatment method is essential to solve the environmental problems as well as to recover those valuable and precious metals that possess high potential for profit. As the natural sources of silver become scarce, it is important to recover silver from wastewater for both environmental and economic benefits.

The currently used methods for treatment of wastewater from the

* Corresponding author: norasikin@cheme.utm.my (N. Othman)
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electroplating industry are coagulation and flocculation, chemical precipitation, membrane filtration, ion exchange, electrochemical treatment, photocatalysis, and adsorption [3-6]. However, the typical concentration of silver in the electroplating rinsing wastewater is very low (5 to 50 ppm) [7], which makes it difficult to be recovered using simple physical and chemical methods. The liquid membrane (LM) technology has demonstrated great potential for the separation of various organic and inorganic compounds [8, 9]. This technology was invented through the combination of liquid-liquid extraction and membrane separation in a single unit. LM processes offer several attractive features including relatively low energy requirement due to its ambient temperature and pressure operation; fast extraction and high efficiency due to the availability of large mass transfer area; the potential for various toxic substances removal even at very low levels; and high selectivity particularly with the use of carrier agents in the LM phase that reacts exclusively with the target solutes [10-12].

The success of this technology mainly depends on its formulation (consist of the carrier, diluent, and stripping agent) and optimization of the processing conditions. A reliable LM extraction system can be achieved via high equilibrium selectivity and capacity as well as a high extraction rate. Previously, a single carrier was used in the extraction system. However, the use of a single carrier in the extraction system suffers from small loading capacity and sluggish phase separation [13,14]. In order to improve the efficiency of ion transport and process, a synergistic extraction system could be developed. Synergism involves the cooperation or interaction of two or more carriers, where the combined effect is larger compared to the sum of single carrier effect. The synergistic extraction can be carried out with the mixture of any type of carriers. For instance, Hu et al. [15] investigated two acidic carriers (Cyanex 272 and Cyanex 923) for the recovery of scandium. Another study reported a synergistic extraction system with basic and neutral carriers of alkylamine and tri-n-butylphosphate [16]. Besides, Homsirikamol et al. [17] investigated the synergistic extraction system using binary mixtures of D2EHPA, TBP, and Aliquat 336. The main advantage of mixed carrier systems is that the reagents are already commercially available thus the time-consuming development of new reagents can be avoided. In addition, an appropriate formulation would be able to reduce the consumption amount of expensive chemicals.

On the other hand, the necessity of green chemistry (to reduce or eliminates the use or generation of hazardous substances in the design, manufacture and application of chemical products) have led to the substitution of organic solvents with environmentally benign green solvents, particularly vegetable oils. The use of such types of green solvents in the organic phase will help in making the industrial processes more environmentally benign while reducing the amount of hazardous wastes at the same time [18,19]. Several studies have reported the use of vegetable oil in liquid membrane formulation. For example, Jusoh et al. [20] incorporated palm oil as a diluent during the extraction of succinic acid. Chakrabarty et al. [21] also used a green diluent for the separation of mercury. In addition, Manna et al. [22] investigated the transportation of catechin antioxidants in a few vegetable oils.

As of now, there have been no presented studies on silver recovery using green synergistic formulation. This study, therefore, attempted to investigate the effects of palm cooking oil incorporation as a diluent in LM formulation. Besides, with the aim to reduce the amount of chemical used, investigations on the effects of individual and carrier mixtures as well as their concentrations were also carried out.

2. Materials and methods

2.1. Chemicals

Silver nitrate solution as feed phase and bis-2-ethylhexyl phosphoric acid (D2EHPA, 95% purity) were purchased from Merck. Refined cooking palm oil was supplied by Lam Soon Edible Oils (Brand: Buruh). Di-2,4,4,-trimethylpentyl mono-thio-phosphinic acid (Cyanex 302, 99% purity), di-2,4,4,-trimethylpentyl phosphinic acid (Cyanex 272, 90% purity), and 2-hydroxy-5-nonylacetophenone oxime (LIX 84-I, 99% purity) were obtained from Sigma Aldrich, Fluka, and Cognis, respectively. All these materials were of analytical grade and used directly as received without further purification.

2.2. Extraction procedures

The LM formulation was developed using a liquid-liquid extraction procedure. In order to determine the potential of the green diluent, an equivalent amount (20 mL) of feed phase (10 ppm silver nitrate solution) and organic phase (Cyanex 302 in palm cooking oil) were combined in a conical flask and agitated by a mechanical shaker at 300 rpm for 18 hours to reach equilibrium. Upon completion of the extraction process, the solution was carefully transferred into a separation funnel and left for 30 min for phase separation. The silver concentration in the aqueous phase formed at the lower layer in the separation funnel was analyzed using an atomic absorption spectrometry (AAS). Meanwhile, the extraction performance was calculated according to the mass balance principle.

In terms of synergism investigation, an equivalent amount (20 mL) of silver nitrate solution was mixed with the organic phase (carrier in palm oil) and agitated at 300 rpm for 1 hour for extraction performance determination. Investigations on the effects of individual and carrier mixtures (Cyanex 302, Cyanex 272, D2EHPA, and LIX 84-I), as well as their concentrations, were also performed using a similar procedure. The phase separation and silver concentration determination were carried out using the aforementioned methods.

2.3. Determination and calculations

The performance of the silver extraction was calculated according to Equation (1), while the distribution ratio (D) and synergistic coefficient (SC) were calculated using Equations (2) and (3).

$$Extraction(\%) = \frac{C_i - C_f}{C_i} \times 100 \tag{1}$$

$$Distribution\ ratio,\ D = \frac{C_{org}}{C_{aq}} \tag{2}$$

$$Synergistic\ coefficient = \frac{D_{mixture}}{D_{carrier} - D_{synergist}} \tag{3}$$

where C_i and C_f are the silver concentrations (ppm) in the aqueous phase before and after extraction. C_{org} and C_{aq} represent the silver concentrations in the organic phase after extraction and aqueous phase after extraction. Meanwhile, $D_{mixture}$ is the distribution ratio of silver obtained from the carrier mixture extraction system, while $D_{carrier}$ and $D_{synergist}$ are the distribution ratio of the silver using the individual carrier.

3. Results and discussion

3.1. Effect of vegetable oil-based diluent

The diluent plays an important role in the LM process and it is a major component in the membrane phase. In most cases, petroleum-based diluent (i.e. kerosene) is used in the LM extraction of silver [23,24], where almost 100% of extraction was reported. The possibility of substituting kerosene with palm oil as the diluent was studied and it was found that the extraction performance was undesirable (7.2%) with only palm oil in the organic phase. This is attributed to the silver ion which is insoluble in the organic phase. Assistance is required for the carrier in order to promote the transport of silver ion from the aqueous phase into the organic phase.

Meanwhile, the extraction of silver significantly increased up to 99.8% with the addition of Cyanex 302 in palm oil, which is comparable with the one obtained using kerosene. These results suggest that palm oil is compatible and can solubilize the carrier. A good diluent must be compatible with the carrier, has low solubility with the aqueous phase (to minimize solvent loss during the extraction process), provide enough density difference from the aqueous (to promote extraction process), low in price, and low in toxicity to prevent pollution. Hence, it can be inferred that palm oil has shown great potential to replace petroleum-based diluent in the organic phase formulation.

3.2. Effect of single carrier concentration

Several studies have confirmed the feasibility of using Cyanex 302 in the recovery of metal ions [1,25-27]. This is due to its characteristics as an acidic carrier capable of extracting metals via a cation-exchange mechanism, in which metals ions are exchanged with hydrogen atoms in Cyanex 302. The metal of interest for the feed solution is silver nitrate ($AgNO_3$). In the aqueous phase, $AgNO_3$ is soluble and exhibits lipophobic properties against the organic phase. The Cyanex 302 dimeric molecules interact with the silver nitrate Ag ions and subsequently form lipophilic complexes.

Figure 1 presents the effect of Cyanex 302 concentration on silver extraction efficiency. The extraction performance can be seen to increase drastically from 28.6% to 95.9% with Cyanex 302 concentration of 0.1 mM to 0.5 mM. This indicates that the amount of Cyanex 302 molecules reacted with silver ions also increased. The results are in agreement with Mane et al. [28], who claimed that an increment in carrier concentration would generally enhance the extraction performance in an ideal extraction system.

However, upon further increase of the Cyanex 302 concentration beyond 0.5 mM, the extraction reaction was found to achieve equilibrium, therefore causing the extraction performance to become plateu. Increasing the Cyanex 302 concentration further showed no substantial effect on the extraction percentage, implying the 'free carriers' were in excess. This result is in line with Noah et al. [2] who indicated that a plateau of extraction efficiency would be obtained at relatively high concentrations of the carrier in the organic phase. With the aim to reduce the amount of expensive chemicals used as well as to develop a synergistic system, 0.1 mM of Cyanex 302 was selected as the favored carrier concentration for the following study.

3.3. Effect of different types of carrier mixture

One way to enhance Cyanex 302 extractive capacities even at low concentrations is by using the synergistic carrier mixture. Synergism is described as a collaboration between two carrier molecules to enhance the extraction performance. For complexion with the metal cation existed in the aqueous feed solution, an anion is required. Therefore, an acidic carrier tends to be highly efficient in separating metal ions via a cation exchange mechanism, in which carrier proton is exchange with metal ions. Figure 2 displays different types of acidic carrier mixtures effect towards silver extraction. The carrier concentration was fixed at 0.1 mM in the mixture system. The results showed that the extraction of silver slightly improved from 28.6% to 29.1% as the mixture of Cyanex 302 and Cyanex 272 was employed.

However, the mixture of Cyanex 302-D2EHPA and Cyanex 302-LIX 84-I provided lower extraction performance compared to the single Cyanex 302. It seems possible for these carrier mixtures to create antagonistic effects towards silver extraction. The formation of hydrogen bonding between these carriers may be responsible for the reduction in silver extraction as explained by Quinn et al. [29]. These results are in accordance with a previous study indicating that an antagonistic effect was observed in the case of Li(I) extraction using a certain carrier mixture [30]. Another author established an antagonistic effect due to the ligand association and decrement in the free main carrier [31].

Meanwhile, as there is only a slight improvement in the extraction performance with the use of carrier mixtures, no significant trend in SC was obtained. Generally, SC > 1 indicates synergistic extraction, SC < 1 implies antagonistic extraction, whereas SC = 1 denotes no synergistic effect [32]. Based on the results, the mixture system of Cyanex 302-Cyanex 272 provided a higher silver extraction of 29.1%, suggesting the presence of synergy in the mixture system. Hence, this carrier mixture is used in the next experiments.

3.4. Effect of mixed carrier Concentration

In order to achieve a maximum extraction performance, a suitable carrier concentration should be formulated as each carrier has a different role in the extraction of silver. Figure 3 illustrates the synergistic extraction of silver in the mixed carrier system of Cyanex 302-Cyanex 272 at an equimolar ratio. It can be seen from the results that the extraction increased with the carrier concentration from 0.1 to 0.3 mM and decreased afterwards. The increase of extraction percentage could be explained by the forward shifting of the reaction according to Le Châtelier's principle as more carriers were added [33].

However, the extraction performance is very much lower compared to that of the single Cyanex 302 carrier as presented in Section 3.2. Besides, the calculated synergistic coefficient value is smaller than 1, thus implying the mixture distribution ratio is less than the sum of distribution ratio for single carriers. Consequently, no or poor synergism occurred in the system as the carriers were in equimolar ratio. A possible explanation for this might be that the carriers were associated with one another in the form of hydrogen bonding [29], therefore reducing the available free carrier to form the complex. This outcome is parallel with that of Atanassova et al. [31] who reported a possible interaction between the two carriers during synergistic solvent extraction of metallic species.

Meanwhile, Figure 4 illustrates the effect of various concentrations of Cyanex 272 on silver extraction in the mixture system. The Cyanex 302 concentration was fixed at 0.1 mM while the Cyanex 272 concentration was varied from 0.1 to 1 mM. Compared to the single Cyanex 302 results, the extraction performance was quite low even during the addition of Cyanex 272 at higher concentration.

This result suggests that the synergistic extraction system with higher Cyanex 272 concentration was unlikely which is confirmed by the SC value of less than 1. This is due to the unique role of each carrier that depends on their surface activity in the organic phase. The carrier with greater surface activity is responsible for "catching" silver ions from the feed phase and transporting them to the organic phase. In the meantime, the carrier with lower surface activity is responsible for the distribution of ions in the organic phase. In the investigation of the synergistic carrier system, Cyanex 302 controls the interface due to its greater surface activity than Cyanex 272. The finding of this study is similar to that observed by Wojcieschowski et al. [34] and Jakubowska et al. [35] in a system involving carrier mixtures.

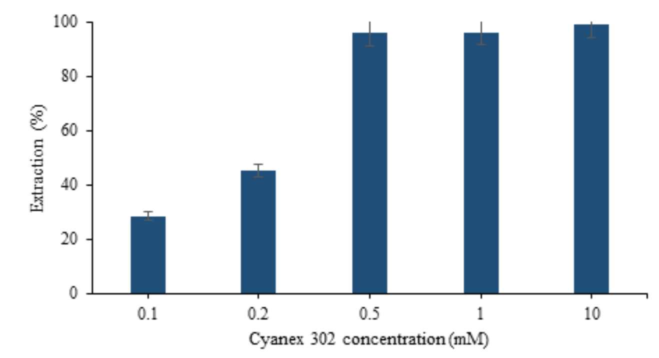

Fig. 1. Effect of Cyanex 302 concentration (diluent: palm oil; feed: 10 ppm silver; agitation speed: 300 rpm; agitation time: 18 hr; temperature: 25ºC).

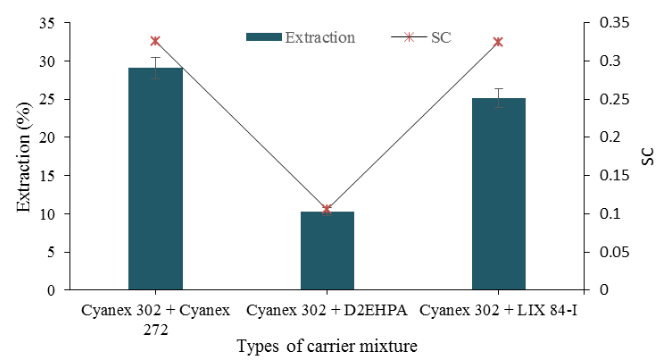

Fig. 2. Effect of acidic carrier mixture (diluent: palm oil; carrier concentration: 0.1 mM; agitation speed: 300 rpm; agitation time: 1 hr; temperature: 25ºC).

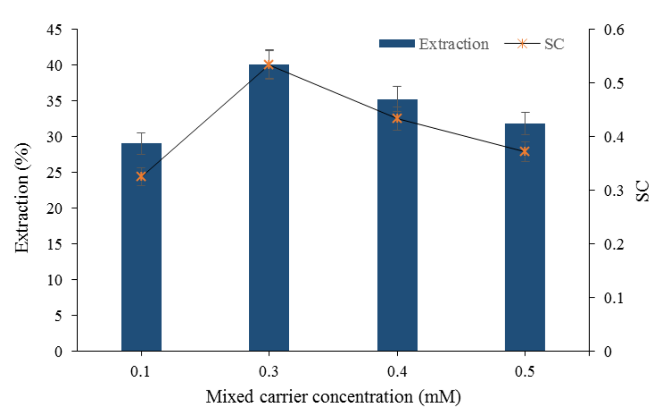

Fig. 3. Effect of Cyanex 302-Cyanex 272 concentration (diluent: palm oil; carrier: equimolar concentration; agitation speed: 300 rpm; agitation time: 1 hr; temperature: 25ºC).

To investigate the number of moles of Cyanex 272 participated in extracting the silver and its composition in the Cyanex 272-silver complex, a graph of log D mixture versus log [carrier] was plotted. The relation of the stoichiometric reaction of silver and Cyanex 272 is shown in Figure 5. The slope of the graph obtained is approximately 0.12, indicating that a very small amount of Cyanex 272 was involved in the complex formation. This finding demonstrated that Cyanex 272 is responsible for the transport of ions through the organic phase.

On the other hand, Figure 6 displays the effect of various concentrations of Cyanex 302 on silver extraction in the mixture system. The Cyanex 272 concentration was fixed at 0.3 mM while the Cyanex 302 concentration was varied from 0.1 to 0.5 mM. It can be seen from the data that in the presence of Cyanex 272, the extraction of silver ion increased dramatically with Cyanex 302 concentration from 0.1 to 0.2 mM and became almost plateau afterward. The extraction performance obtained using 0.2 mM Cyanex 302 and 0.3 mM Cyanex 272 was 98%, which is twice the performance of 0.2 mM Cyanex 302 (45.4%). Although the extraction performance was similar to the one obtained by 0.5 mM Cyanex 302 (96%), it should be noted that the single extractant system was conducted in 18 hours, while the synergistic extractant system was conducted in only 1 hour. Hence, the extraction performance was enhanced by the synergistic mixture of 0.2 mM Cyanex 302 and 0.3 mM Cyanex 272. In addition, a very high SC value was obtained, indicating the occurrence of the synergistic effect within the system.

Meanwhile, Figure 7 shows the plot of log D mixture vs. log [Cyanex 302] at a specified Cyanex 272 concentration. The slope of the graph is 4.1, suggesting 4 moles of Cyanex 302 were involved in the extraction reaction. The main carrier typically provides slow kinetics, based on the basic theory of kinetic, while another carrier designated as the synergist gives fast kinetics and accelerate the extraction process. Thus Cyanex 272 and Cyanex 302 served as a carrier and synergist respectively in this study.

4. Conclusions

In summary, a green synergistic formulation for silver recovery was successfully carried out. Palm oil possesses high potential as a substitute for the conventional petroleum-based diluent. The mixture carrier of Cyanex 302-Cyanex 272 synergistically improved the silver ions extraction. At a concentration of 0.2 mM Cyanex 302 and 0.3 mM Cyanex 272, more than 98% of silver was extracted with SC of 62.7. Based on the investigation of the individual and carriers mixture results, it can be inferred that Cyanex 272 and Cyanex 302 serves as a carrier and synergist, respectively. Therefore, the findings of this study can be used as a basis in the development of the liquid membrane application for silver recovery from industrial wastewater.

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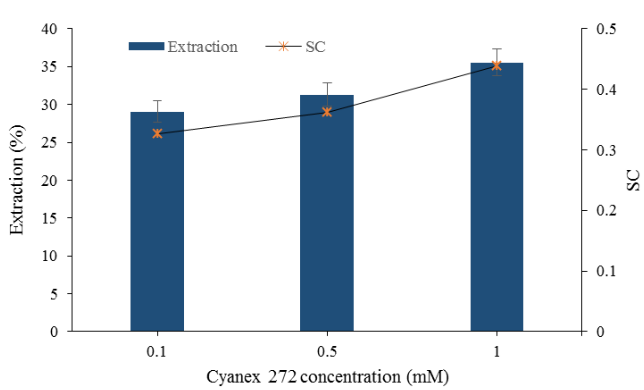

Fig. 4. Effect of Cyanex 272 concentration in mixture system (diluent: palm oil; fixed carrier: 0.1 mM Cyanex 302; agitation speed: 300 rpm; agitation time: 1 hr; temperature: 25ºC).

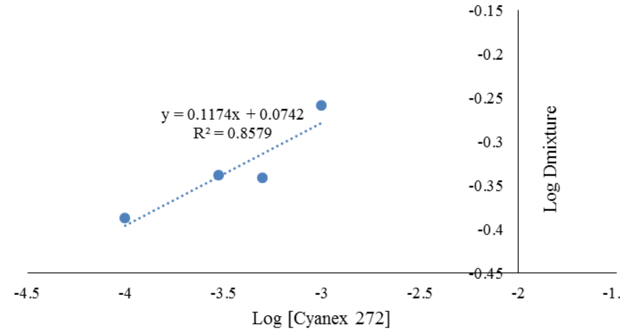

Fig. 5. Effect of Cyanex 272 concentration on the extraction of silver at a fixed Cyanex 302 concentration.

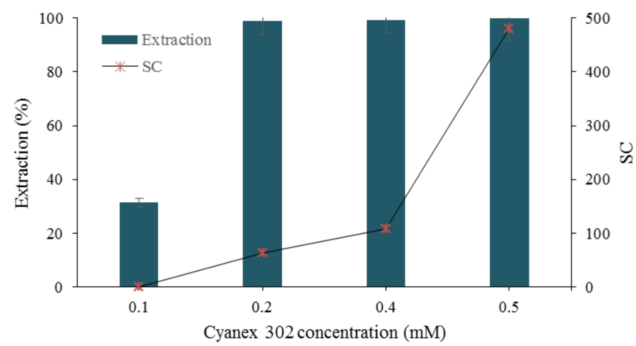

Fig. 6. Effect of Cyanex 302 concentration in mixture system (diluent: palm oil; fixed carrier: 0.3 mM Cyanex 272; agitation speed: 300 rpm; agitation time: 1 hr; temperature: 25ºC).

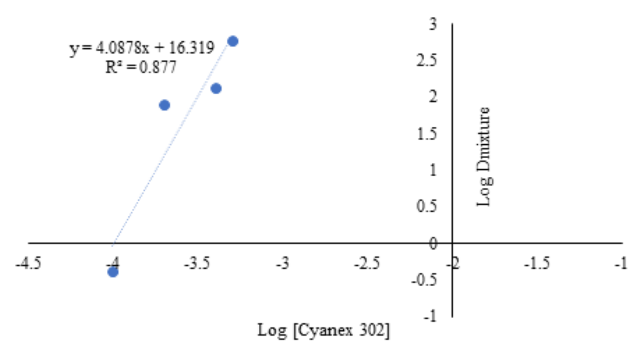

Fig. 7. Effect of Cyanex 302 concentration on the extraction of silver at a specified Cyanex 272 concentration.

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